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Effect of Particle Size of Chemical Mechanical Polishing Slurries for Enhanced Polishing with Minimal Defects

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In this study the effects of oversize particle contamination in chemical mechanical polishing (CMP) slurries were investigated on the silica CMP process. The limits of light scattering technique were established in detecting coarse particles in a commercial silica CMP slurry using two different methods. The detection limits were set by observing the shift in particle size distribution curve or by the appearance of an additional peak in the particle size distribution curve of the baseline slurry when a known amount of coarser particles were added to it. Simultaneously, polishing tests were conducted by spiking the base slurry with coarser sol-gel silica particles at the established detection limits. It was observed that the contamination of larger particles not only created surface damage but also changed the material removal rate. The mechanism of polishing in the presence of larger size particles is discussed as a function of particle size and concentration.

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Chemical mechanical polishing (CMP) is a widely used technique for the planarization of metal and dielectric films to accomplish multilevel metallization (MLM). The rapid advances in the microelectronics industry require a decrease in the size of microelectronic devices,[1] and the fabrication of these small feature devices without defects requires significant improvements in the CMP process.

The success of CMP operations depends on the rate of material removal and the quality of the surface finish. Optimal removal rate and smooth surface finish are achieved by the synergistic effect of the chemical and mechanical forces encountered in the CMP process. It has been demonstrated that in oxide CMP, the chemical content of the polishing slurry is responsible for enabling erosion, whereas mechanical forces help achieving the required planarization and uniformity.[2] The erosion action in CMP is mostly provided by the submicrometer size abrasive particles as they flow in between the pad and the wafer surface under applied pressure. The magnitude of the mechanical action is in turn determined by the concentration and the size of the abrasive particles among other things. Therefore, it is important to know the optimal abrasive size distribution to enhance the removal rate without damaging the wafer surface. Cook reported that the surface damage was less when monosized slurries were used for polishing.[3] However, in practical applications slurries with wide size distributions are often used. A small fraction of coarse particles in these slurries can create deformation on the wafer surfaces, resulting in defective devices. Hence, the detection and removal of oversized particles at small concentrations is critical for efficient CMP operations.

Structural damage on wafer surfaces can be classified as scratches, pits, delamination of film interfaces, and the introduction of chemical or particulate impurities and changes in the chemical or physical structure of the film.[4] Among these, the major defects created by the contamination of oversize particles are the formation of microscratches and pitting of the wafer.[5] The importance of detecting the coarse fraction in CMP slurries has recently received added attention, as the reduction of surface defects has become more important due to the continuously decreasing size of the critical dimensions. It was shown previously by Pohl and Griffiths that the static laser light scattering technique was capable of detecting the 1.0 μm particles in 0.4 μm size baseline slurry and could be used to monitor the removal of the unwanted coarser sized abrasive particles from the slurry.[6] The impact of the slurry particle distribution on defects and the vulnerability of the post-CMP cleaning process was also studied by Nagahara *et al.* using Accusizer 770.[7] This instrument was found to be sensitive in detecting the particles greater than 1 μm size. A direct correlation was reported between the amount of the large slurry particles and the particles remaining on the wafer. In another study, the sensitivity of acoustic spectroscopy technique was investigated by Dukhin and Goetz in detecting the coarser particles in a fine particle size slurry.[8] They reported that the detection limit could be as low as a single 1.0 μm particle per 100,000 particles of 0.1 μm size. Additionally, it was assumed that larger particles would not form appreciable defects at the established detection limit, however, no supporting polishing test results were presented.

In the present investigation, the limits of a static light scattering technique in detecting a small number of larger particles in a commercial CMP slurry were established. Simultaneously, polishing tests were conducted at the established detection limits to analyze the changes in surface topography and removal rate in the presence of the coarser particles.

Experimental

The particle size analyses were conducted by using a Coulter LS 230 light scattering instrument with small volume module. Rodel 1200 commercial fumed silica slurry with a mean particle size of 0.14 μm was used as the baseline CMP slurry (supplied by Rodel® Inc.). This slurry was spiked with sol-gel silica particles of 0.5, 1.0, and 1.5 μm sizes that were obtained from Geltech Corporation.

To establish the detection limits of the light scattering technique, the 20 mL slurries of Geltech silica particles were prepared at 5 wt % solids at pH 10.5 using the Rodel 1200 slurry supernatant to preserve the original chemical composition of the baseline slurry. The base Rodel 1200 slurry was also diluted from 12 to 5 wt % solid concentration using its own supernatant. The mean particle size of the baseline slurry was found to be the same after the dilution. The Geltech sol-gel silica slurries were sonicated for 40 to 60 min in an ultrasonic bath until all the agglomerates were broken and the mean particle size matched the particle sizes observed by scanning electron microscopy (SEM) analysis. The coarser size Geltech slurries were mixed with the base slurry using a microliter syringe with a total volume of 500 μL. The spiked slurries were sonicated for an additional 15 min and then fed into the analyzer. The measurements were conducted by using pH-adjusted water (pH 10.5) in Coulter LS 230 to prevent the agglomeration of particles during the measurements.

The polishing tests were performed on p-type silicon wafers on which a 1.5 μm thick SiO_2 layer had been deposited by plasma enhanced-chemical vapor deposition (PECVD) (supplied by Silicon Quest International). The 8 in. wafers were cut into square samples of 1.0 $\times$ 1.0 in. and a Struers Rotopol 31 tabletop polisher was used for polishing with integrated circuit (IC) 1000/Suba IV stacked pads supplied by Rodel Inc. The downforce was 7.0 psi (492 g/cm^2), and

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the rotation speed was 150 rpm both for the pad and the wafer. The thickness of the oxide film on the wafers was measured by the spectroscopic ellipsometry method before and after polishing to calculate the removal rate. The slurry flow rate was 100 mL/min. The polishing tests were conducted with 50 mL slurries for 30 s at a solid concentration of 12 wt %. Atomic force microscopy (AFM) technique was used for the surface roughness and deformation analysis of the polished wafers. Polishing tests were repeated five times using the baseline and spiked slurries. AFM analyses were conducted by taking five micrographs at the central parts of the polished wafers. The reproducibilities of the surface roughness results were in the range of the reported standard deviations. The micrographs presented in the figures were selected based on the clarity of the observed defects such as pit formations or scratches on the surfaces.

Results and Discussion

Detection of coarser particles in CMP slurries.—The limits of the static light scattering technique in detecting a small amount of coarse particles in CMP slurries were established using two different methods. The first method was based on detecting a shift in the mean diameter and size distribution curve of the baseline slurry, as it was gradually spiked with increasing concentration of the larger size particles. The mean size of the spiked slurries increased with increasing number of the coarser particles. However, until a critical concentration of coarser sizes was added in the baseline slurry, the change in mean size was not statistically significant. When the critical concentration was reached, both the mean diameter and size distribution curve of the spiked slurries were observed to shift significantly. Therefore, this particular concentration of the coarser particles was set as the "curve shift detection limit." The second method was based on the appearance of a second peak in the size range of the spiked particles. Above a certain concentration of the spiked coarser particles, the Coulter LS 230 software started to fit the data to a multimodel distribution, representing the coarser particles as an individual peak. The concentration of larger size particles required to form an extra peak was defined to be the "double peak detection limit."

Figure 1 shows the change in the particle size distribution of Rodel 1200 slurry as a function of Geltech 1.5 μm coarse particle addition. The mean particle size of the Rodel slurry was 0.14 μm, and it originally exhibited a bimodal distribution with the main peak at 0.1 μm and a second peak at 0.5 μm. The analysis conducted on the Rodel slurry by using Accusizer 780, which is a number counting technique, also showed the presence of the larger particles. It is suspected that the fused silica chains in the Rodel slurry entangle in nonspherical shapes and detected as larger particles due to the scattering through the longer dimension. As the 1.5 μm Geltech particles were gradually added into the baseline Rodel slurry, a statistically

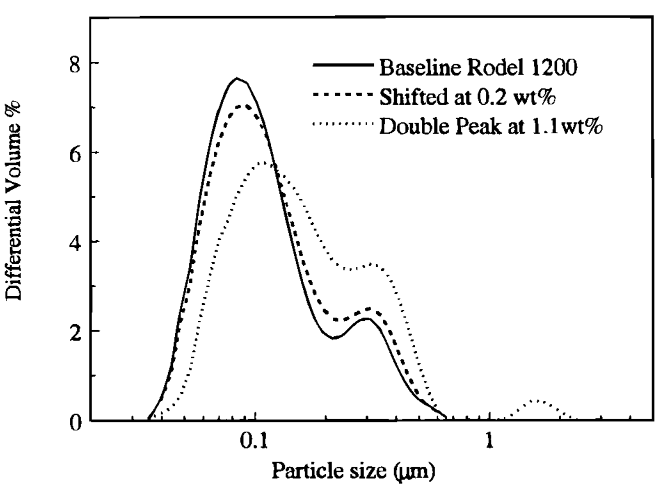

Figure 1. Particle size distributions for Rodel 1200 slurry spiked with Geltech 1.5 μm particles.

Table I. Detection limits of Coulter LS 230 for Rodel 1200 slurry spiked with 0.5, 1.0 and 1.5 μm Geltech particles.

Feed slurry: Rodel 1200 (η = 0.14 μm)

Detection limit		Coarse particles 0.5 μm	1.0 μm	1.5 μm
Double peak	by weight	N/A	1.3%	1.1%
	by number coarse:fine	N/A	1:13,900	1:84,000
Curve shift	by weight	1.1%	0.3%	0.2%
	by number coarse:fine	1:4,100	1:60,800	1:460,000

95% Confidence interval: ±2%.

significant shift of the mean particle size was observed at 0.2 wt % addition of the coarser fraction. When the amount of coarser particles was increased further, an individual peak was detected at the 1.5 μm size range at a concentration of 1.1 wt % representing the double peak detection limit. Table I summarizes the detection limits of Coulter LS 230 for the Rodel slurries spiked with 0.5, 1.0, and 1.5 μm Geltech particles. Since the original Rodel slurry had exhibited a peak at the 0.5 μm range, the second peak detection could not be established for the slurries spiked with 0.5 μm particles.

It was observed that the sensitivity of coarse particle detection improved as the difference in the mean particle size of the base slurry and the spiked coarser particle size increased. In addition, the curve shift method was able to detect the larger particles at relatively lower concentrations. It was found that, one 1.5 μm particle for every 460,000 Rodel particles (0.14 μm) can be detected using the curve shift detection limit. This ratio was 1:84,000 using the double peak detection protocol. However, the curve shift method can be used only if there is information available about the characteristics of the baseline slurry (such as mean particle size and standard deviation of the distribution).

Surface roughness and critical damage analysis on the wafer surfaces in the presence of coarser particles during CMP.—To examine the surface morphology and defect formation in the presence of coarser particles, polishing tests were conducted with the slurries prepared by mixing Geltech coarse particles into the baseline Rodel slurry at the established detection limits of the static light scattering technique. Subsequently the polished wafer surfaces were analyzed with atomic force microscopy (AFM). In previous work, the effect of particle size on oxide CMP was investigated by using 0.2, 0.5, 1.0, and 1.5 μm monosize slurries prepared by Geltech silica powders at various solid loadings.[9] Significant defect formation on the wafer surfaces was detected with the slurries made of 0.5 μm size particles. Both the surface roughness (rms) values and the surface deformation increased with the increasing size and the concentration of the abrasive particles. The morphology of the defects was often observed as pits rather than scratches.

Figure 2 shows the three-dimensional AFM pictures (5 × 5 μm) for the surfaces polished with the base Rodel 1200 slurry and the slurries spiked with 1.1 wt % 0.5 μm (statistical shift detection limit) and 1.5 μm (double peak detection limit) Geltech particles. It is clearly seen in Fig. 2a that the polishing with Rodel slurry yielded acceptable polishing results. The maximum depth of surface damage (R_{max}) was less than 3 nm and the rms roughness was 0.5 nm. However, the slurry spiked with the coarser particles at 1.1 wt %, resulted in surface deformation, which increased with the increasing particle size. Pitting and scratch formation was observed on the oxide film as seen on Fig. 2b and c. R_{max} values reached 15 and 40 nm with 0.5 and 1.5 μm size particles, respectively. The formation of the pits may be explained by the increased chemical activity at the contact points of the coarse particles with the wafer under the applied pressure. It has been suggested for tungsten polishing that the abrasive

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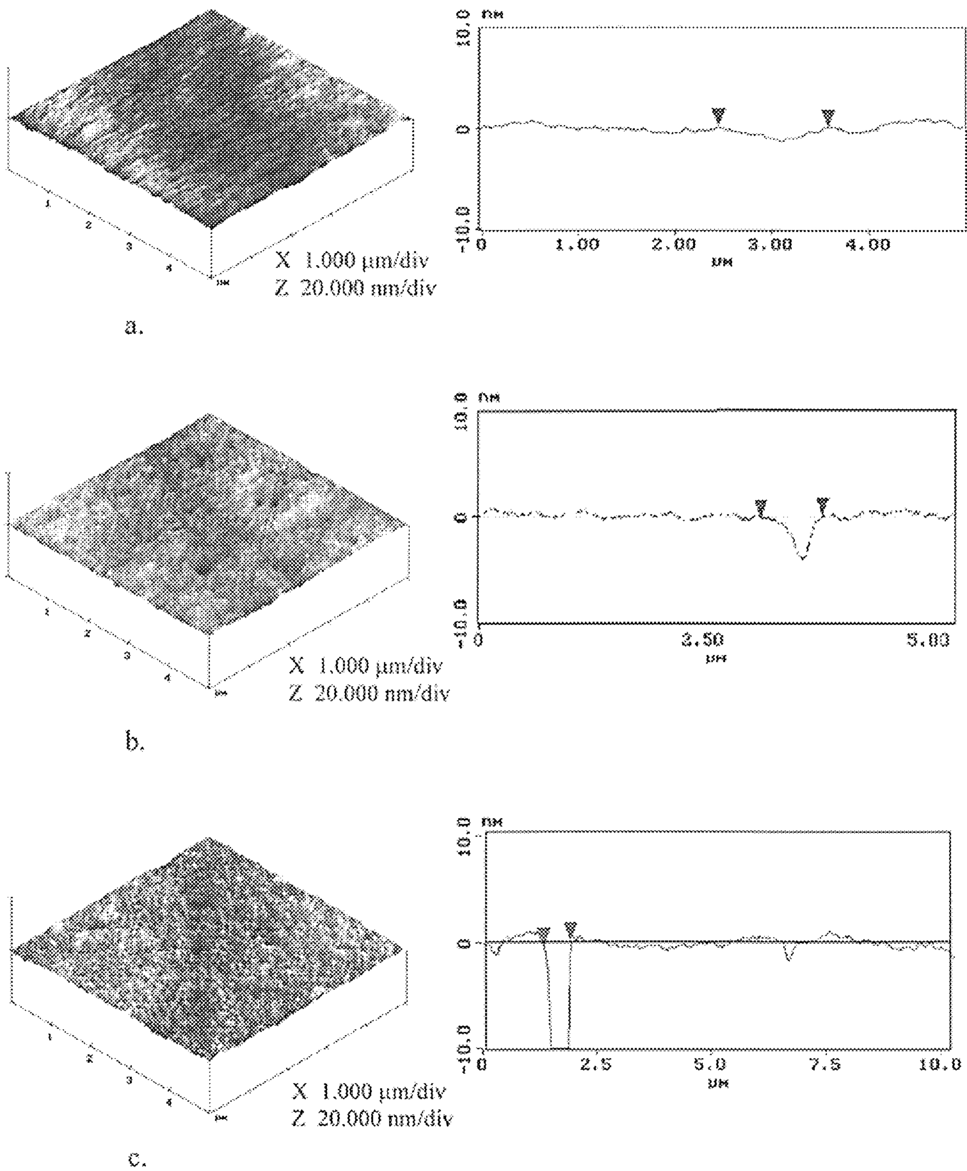

Figure 2. (a, top) AFM picture and cross section for the wafer polished with Rodel 1200 baseline slurry. (b, middle) AFM picture and cross section for the wafer polished with Rodel 1200 slurry spiked with 1.1 wt % Geltech 0.5 μm particles. (c) AFM picture and cross section for the wafer polished with Rodel 1200 slurry spiked with 1.1 wt % Geltech 1.5 μm particles.

particles and their surface chemistry may cause local variations in process temperature and polish rate.[10] It was also reported that in addition to causing mechanical abrasion the abrasive particles also increase the local temperature due to friction resulting in enhanced chemical action.

Figure 3 shows the surface and cross-sectional analyses of the wafers polished with Rodel slurries spiked with 1.0 μm particles at curve shift (0.3 wt %) and double peak (1.3 wt %) detection limits. The effects of increasing coarse particle concentration are evident in these pictures. At 0.3 wt % concentration of 1.0 μm particles (Fig, 3a), the surface was relatively smooth, but scratch formation was detected. This may be due to the relatively higher pressure per particle at low concentrations (0.3 wt %) of coarse particles compared to the higher concentrations (1.3 wt %). The calculations showed that the width of the detected scratch matched the dimensions of one that could be formed by a 1.0 μm particle at the measured depth. As the concentration of 1.0 μm particles increased to 1.3 wt % (Fig. 3b), the surface roughness increased and local deformations and severe pitting started. This may be caused by the increasing interaction of coarser particles with each other and with the Rodel particles on the wafer surfaces at higher concentrations. This mechanism is suggested by the widths of the pits being significantly larger at higher concentrations of 1.0 μm particles than the lower concentrations, as can be seen on the cross sections of Fig. 3. It is believed that at low concentrations of coarser particles the particle-particle interaction is much less, and the oversize particles tend to create more scratches than local deformations on the surface under the effect of high pressure.

Table II summarizes the quantitative results of the polishing tests. It is clear that the surface roughness and the degree of surface damage (R_{max}) increased with the increasing size and concentration of coarser particles in the spiked slurries. The standard deviations of rms roughness between similar samples were also greater at higher concentration of coarser particles, indicating the increase in the overall surface damage. In general, the presence of the coarser particles resulted in a significant increase in the surface roughness and surface deformation mostly by causing pit formation on the wafer surfaces.

Material removal rate in the presence of coarser particles.—The removal rates obtained with and without spiking the Rodel slurry with the larger particles are shown on Fig. 4. The oxide removal rate of the baseline Rodel 1200 slurry was 10,000 Å/min. As the coarser particles were spiked into the Rodel slurry, the removal rate decreased consistently, except for the slurries spiked with 1.5 μm particles at 1.1 wt % concentration for which the removal rate was 10,300 Å/min. Table II also summarizes the results of the material removal (polishing) rates.

The polishing process is generally explained by the Preston equation.[11] This equation accounts for the pressure and the linear veloc-

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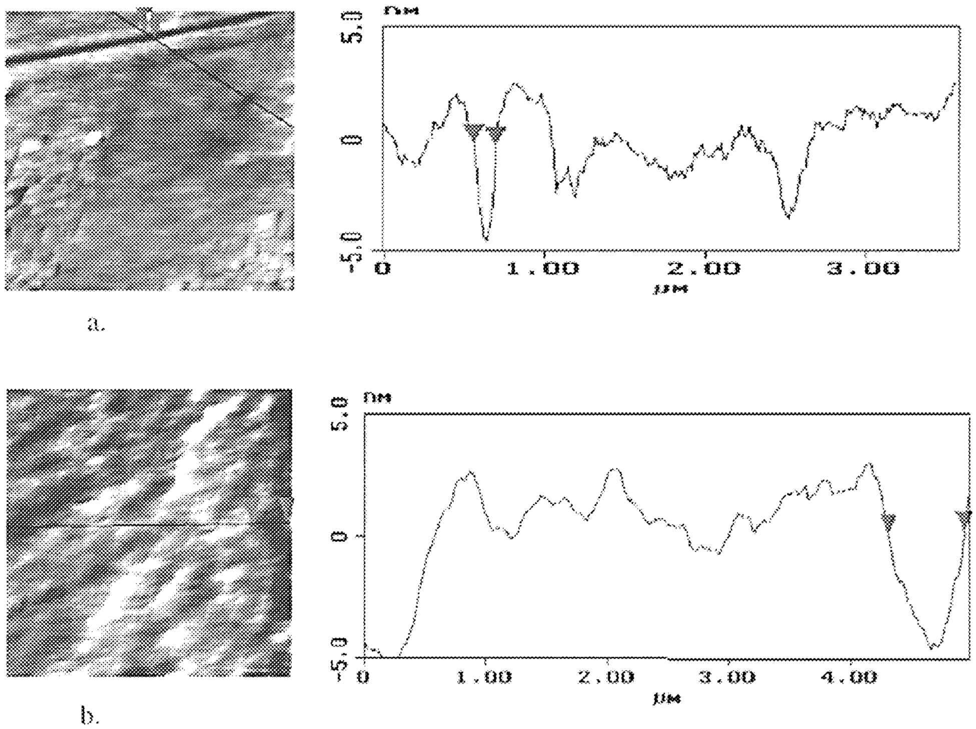

Figure 3. (a, top) Cross-sectional analysis for the wafer polished with Rodel 1200 slurry spiked with 0.3 wt % Geltech 1.0 μm particles (statistical shift detection limit). (b, bottom) Cross-secional analysis for the wafer polished with Rodel 1200 slurry spiked with 1.3 wt % Geltech 1.0 μm particles (double peak detection limit).

ity of the pad relative to the work piece during the polishing operation. All the other variables such as abrasive properties, slurry chemistry, and tribological interactions at the wafer-slurry-pad interface are assumed to be accounted for in the Preston coefficient.[12,13] However, for the purposes of the present study it is important to know the effect of abrasive size on polishing in order to explain the changes in the removal rate for the slurries containing a small amount of coarser particles. Although this subject has been investigated for various CMP operations, the reported results are often contradictory stating that the polishing rate is independent, increasing, or decreasing with the slurry particle size.[3,14-16] This discrepancy is believed to be due to the selected particle size range, the polishing pad used, and the varying experimental conditions affecting the slurry chemistry and tribological interactions at the wafer-slurry-pad interface.

The polishing mechanism for the silica/silica system has been recently studied by our group as a function of particle size and solids concentration.[9] In this study, Geltech silica powders at 0.2, 0.5, 1.0, and 1.5 μm sizes were used under the same experimental conditions as in the present study. The slurry solids concentrations were varied between 2 and 15 wt %, and the slurry pH was adjusted to 10.5. The results of monosize particle polishing indicated a change in the predominant polishing mechanism as a function of the particle size and solid loadings. The removal rate was measured to increase with the increased solid loadings for the 0.2 μm slurries. On the other hand, for the 1.0 and 1.5 μm size slurries a consistent decrease was observed in the polishing rate as a function of the solid loadings. In agreement with these results, an inversion was detected in the material removal rate with 0.5 μm size slurries. Initially there was an increase in removal rate up to 5 wt % solids concentration followed by a decrease at higher solids loading. These results were explained based on two mechanisms; namely, contact area mechanism and indentation-based mechanism that were first introduced for tungsten polishing.[17] It was discussed that using small particle sizes the contact area mechanism was predominant and as the slurry particle size increased the indentation based mechanism became more significant. The mathematical expressions for the solid loadings and particle size dependence were derived by Singh *et al.*[17] from Brown's[18] expression for penetration depth of an abrasive particle.

According to the contact area mechanism, the material removal rate is proportional to the total contact area between the abrasive particles and the wafer surface. This mechanism is thought to enhance the chemical activity on the surface due to the interactions between the particle and film. The total contact area will increase with the increasing concentration and decreasing particle size. It is clear that at higher solid loadings there will be more particles in contact with the substrate and hence a greater removal rate may be expected. However, at a fixed concentration of solids, the number of particles will increase as the particle size decreases, which explains the

Table II. Summary of the polishing results for the Rodel 1200 slurry spiked with 0.5, 1.0, and 1.5 μm Geltech silica particles at the established detection limits of Coulter LS 230.

Detection limit	Coarse particle size (μm)	% Coarse particles (by weight)	Removal rate (Å/min)	rms (nm)	R_{max} (nm)
	Baseline	None	9993 ± 380	0.50 ± 0.15	3
Double peak	1.0	1.3	7913 ± 519	1.76 ± 1.47	20
	1.5	1.1	10308 ± 594	1.33 ± 1.02	40
Curve shift	0.5	1.1	8797 ± 160	1.79 ± 1.57	15
	1.0	0.3	7874 ± 391	1.00 ± 0.19	15
	1.5	0.2	6771 ± 494	1.31 ± 0.66	20

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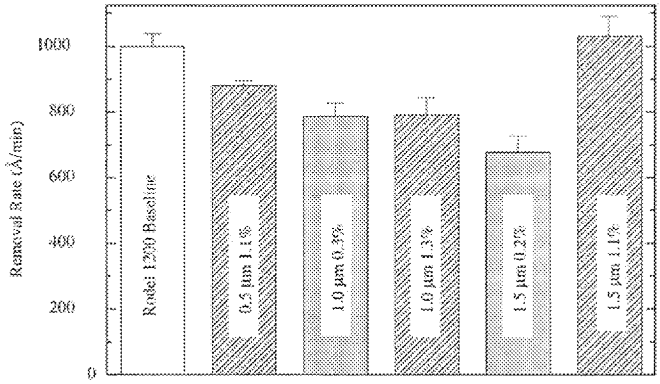

Figure 4. Polishing rates for the baseline and spiked Rodel 1200 slurries. (□) Baseline Rodel 1200 slurry. (▒) Rodel 1200 slurry spiked with Geltech coarse size particles at high concentrations (~1.0% by weight). (□) Rodel 1200 slurry spiked with Geltech coarse size particles at low concentrations (~0.25% by weight).

inverse relationship between particle size and contact area. The total contact area, A, can be expressed as a function of the abrasive concentration C_o, and particle diameter Φ as shown in Eq. 1

$$A \propto C_0^{1/3} \cdot \phi^{-1/3} \quad [1]$$

The indentation-based mechanism explains the material removal as a result of indentations created by the abrasive particles. This is mostly a mechanical mechanism creating abrasion on the surface. The total indent volume, V, is inversely proportional to the concentration of particles and directly proportional to the particle size as shown in Eq. 2. The indentation of the particles increases as the pressure per particle increases, which is possible by decreasing the number concentration of particles on the surface. Hence, the total indent volume is higher at low particle concentrations and also when larger particles are used at a fixed concentration

$$V \propto C_0^{-1/3} \cdot \phi^{4/3} \quad [2]$$

Figure 5 schematically depicts the proposed mechanism for material removal for the slurries spiked with the coarser particles. It is suggested that the coarser particles tend to hold the wafer away from the pad reducing the pressure on the smaller size abrasive particles resulting in decreased interaction of the Rodel particles with the substrate. The decrease in the total contact area between the wafer and the abrasive particles results in reduced removal rates. On the other hand, the total indent depth of the coarser particles is higher as explained by the indentation-based mechanism. This increase in the particle indent for the spiked slurries at higher concentrations of the coarser particles in turn results in higher removal rates albeit at the expense of the formation of pits and higher surface roughness. However, the validity of the proposed mechanism needs to be confirmed for different pad types (other than Rodel IC 100/SUBA IV stacked pad used in this study).

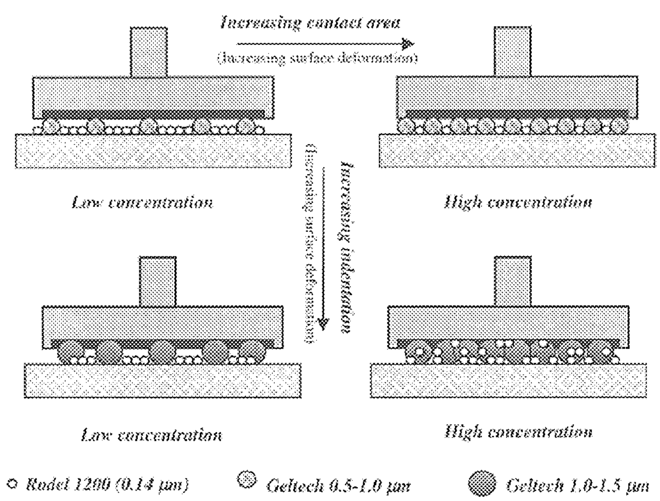

Figure 5. Change in polishing mechanism for the Rodel slurries spiked with Geltech coarser particles based on the contact area and indentation based mechanism.

It is also very well known that at high pH values and temperatures silica solubility is higher.[4] As explained previously it is believed that for the spiked slurries the applied load is carried by the larger size particles more than the smaller sizes resulting in higher local pressure and temperature on the wafer surfaces in contact with the coarser particles. The observed increase in the depth of surface deformations with increasing size and concentration of the coarser particles may also be explained based on the higher local dissolution of the silica under these conditions.

The findings reported in this study are consistent with the argument that the contact-area-based mechanism is dominant at the small particle sizes and the indentation mechanism takes over as the abrasive size increases. It is believed that at a particular population of larger particles both the contact area and the indentation based mechanisms become equally effective as seen for the 1.5 μm particles at 1.1 wt % concentration. At this concentration of the 1.5 μm particles the total contact area is relatively large compared to 0.2 wt % concentration but the indentation of the particles on the surface is also considerable. Consequently, the removal rate obtained at high concentrations of coarser particles approaches the original removal rate of the baseline Rodel slurry. Yet, the critical surface damage occurring under these conditions (as seen on Fig. 2c) indicates that this is not an optimal size distribution for the CMP slurries.

Conclusions

The limits of the light scattering technique in detecting a small number of coarser particles in a commercial CMP slurry were established using the Coulter LS 230 particle size analyzer. The results showed that the detection limits of the light scattering technique could be as low as parts per million level (two 1.5 μm particles for a million Rodel 1200 particles (0.14 μm) using the statistical shift method). The double peak detection method, on the other hand, was able to detect the coarser particles (five to ten times larger than the base slurry particles) at approximately 1 wt % concentration of the total solids in the slurry.

The results of the polishing tests showed that the presence of coarser particles tends to create critical defects on the oxide film and change the polishing mechanism. The surface damage increased with the increasing size and the concentration of the coarser size particles in the baseline slurry. It was also observed that the polishing mechanism may vary based on the size and the concentration of the coarser particles. These findings indicate the critical need to detect the coarser particles at much lower concentrations and find effective ways of removing them from the CMP slurries in order to obtain optimal polishing results.

Acknowledgments

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